Δ[13] C ISOTOPE AS PRECURSOR TO RECONSTRUCT PALEO-VEGETATION TYPE AND DEPOSITIONAL ENVIRONMENTS OF MID-SIWALIK SEDIMENTS OF NW HIMALAYAN FORELAND BASIN, PAKISTAN

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ABSTRACT: Using an elemental analyzer-isotope ratio mass spectrometer (EA-IRMS), the organic matter extracted from sandstone samples of the mid-Siwalik Dhok Pathan Formation exposed on the western limb of the Makerwal anticline in the Surghar-Shingar Range of the NW-Himalayan foreland Fold-and-Thrust-Belt was examined for δ^{13}C isotope. These molasse sediments have revealed significant information about the paleo-vegetation type and their depositional environments in the sandstone facies. Their average δ^{13}C isotope value calculated through EA-IRMS is -26.56‰, which corresponds to cold growing environment C_3 type vegetation. The δ^{13}C isotope findings range from -24.50 to -28.43‰. The δ13C isotope data showed that C_3 biomass dominated in the ecosystem throughout the Miocene particularly in the studies part of foreland basin. The coaly-to-semi coaly phytoclasts are classified as vitrinite-huminite based on their morphology obtained through back scattered imaging on scanning electron microscope (SEM). These phytoclasts had been contributed by terrestrial vegetation and designated as Type-III kerogen. The SEM study confirms the development & replacement of diagenetic frambiodal pyrite, which advocate the deposition under reducing conditions.

Key words: Stable carbon isotope, sandstone, Paleo-vegetation, Siwalik, Depositional Environs.

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INTRODUCTION

The climatic variations on local and regional scale are well recording in sediments/sedimentary rocks, which enfold imperative records of paleo-environmental conditions continually. Resultantly, this change in climate can cause modification in vegetation types. The humidity and associated temperatures of the environment/climate are generally connected with the growth of terrestrial land plants (Ali *et al*., 2020b). Numerous research has shown that the kind of vegetation may be directly impacted by local climatic fluctuations (Takahara *et al*., 2010; Lim *et al*., 2013; Hyun *et al*., 2015). There may be a large-scale worldwide link between the related vegetation type and climatic fluctuation (Litwin *et al*., 2013). By interpreting differences in organic proxies, such as stable isotopes of hydrogen, oxygen, nitrogen, and carbon (δ^{13}C) in various ecological/geological environments, one can determine the alteration in paleo-vegetation and paleo-climate and forecast the paleo-environmental conditions (Yamamoto *et al*., 2010; Zhang *et al*., 2010; Zech *et al*., 2012, Hyun *et al*., 2015).

The paleo-environmental changes can be deduced by analyzing the isotopic concentrations present in organic matter (OM) preserved in sediments/sedimentary rocks (Kuramoto and Minagawa, 2001; Omura and Hoyanagi, 2004; MA *et al*., 2009; Milligan *et al*., 2010; Kohn, 2010; Prentice *et al*., 2011; Schubert and Jahren, 2012; Diefendorf *et al*., 2015; Khan *et al*., 2015; Hatem *et al*., 2016; Kohn, 2016). Higher rates of sedimentation and short-term depositional processes are both well-preserved in the sediments (Meyers, 2003; Ahmad and Davis, 2017). The composition of organic matter and rate of accumulation are being affected by both in-situ and upland deposition basinal conditions (Killops and Killops, 2013; Ahmad and Davis, 2017). The source of organic matter accumulated during deposition of sediments can be differentiated by analyzing values of δ^{13}C (Shanahan *et al*., 2013; Khan *et al*., 2015).

At the depositional stage of sediments, the amount and composition of organic matter replicate the frequency and nature of biota those endure within the sedimentary basin. The information about distribution and vegetation-types can be figured out from isotopic values of organic matter. These isotope studies are the instructive methods for reconstructing the paleo-environmental conditions (Lü *et al*., 2000; Wang *et al*., 2003; Ahmad and Davis, 2017).

The source of organic matter can be implicit from isotopic organic proxies such as C/N ratio, total organic carbon and stable carbon isotope values. The proxies are also useful to identify the organic carbon sources within the sediments (Ahmad and Davis, 2017; Ali *et al*., 2019). The geochemical evidence of the sediments is, however, manipulated by a number of factors, including the presence of organic matter, the pace of sedimentation, and diagenesis (Tyson, 1995). Research on long-term fluctuations in the environmental record is crucial for projecting future changes globally (Matsumoto *et al*., 2012; Ahmad and Davis, 2017). The chemo-stratigraphy, paleo-oceanography, characterization of organic matter, and correlation/evaluation of oil source rocks have also been studied by analyzing isotopic ($\delta^{13}C$) concentrations (Hayes *et al*., 1989; Fontugne and Calvert, 1992; Hollander *et al*., 1993; Tyson, 1995).

The early diagenetic processes such as fluvial input distance; redox state of sediments; oxygenation; trophic state & salinity; burial/thermal maturity; water column depth; bio-chemical reactivity; amount & type of hydrocarbon can be scrutinized by organic matter characterization present in the sedimentary records (Tyson, 1995). It is a practical tool for the modeling of carbon cycle by differentiating between autochthonous & allochthonous organic matter as well.

The drainage networks and paleo-climatic conditions have recorded significant information about Himalayan orogeny, which is well preserved in the Neogene molasse sediments called Siwalik Group. These sedimentary rocks are well preserved in Himalayan foreland basin (Najman, 2006). The rocks of Siwalik Group are well preserved and out-cropped in many areas' country-wide including Salt Range; Bannu Basin; Suleiman Basin; Kohat-Potwar Plateau and Kirthar-Fold-and-Thrust belt etc. (Ullah *et al*., 2009; Shah and Hafeez, 2009; Ali *et al*., 2019). The research region is located in the Trans Indus Salt Ranges, on the western face of the Makerwal Anticline, or Surghar-Shingar Range (Fig. 1a). While the rocks exposed in the Trans Indus Salt Ranges have received relatively little attention, many researchers have thoroughly examined the molasse sediments that are exposed in the Kohat-Potwar Plateau and Suleiman Ranges (Barry *et al*., 1985; Quade *et al*., 1989; Shah and Hafeez, 2009; Ullah *et al*., 2009; Ali *et al*., 2019, Ali *et al*., 2020b etc.). Due to easy access to known coal reserves that have been mined for millennia, the eastern limb of the Makerwal Anticline was the major focus of earlier study (Ali *et al*., 2018b). Enormously, a little scientific data is available about paleo-environment / depositional environments; the source & characterization of organic matter preserved in mid-Siwalik Dhok Pathan Formation of Makerwal Anticline; also called Shingar-Surghar Range (SSR). The primary objective of this study is the utilization of $\delta^{13}C$ data to re-construct the paleo-vegetation type, paleo-climatic conditions and characterization of source of organic matter. It will be helpful in understanding the paleo-climatic variations and associated vegetation-type in the studied part of the basin.

Geological Setting: A flexural curvature was formed in the south of rising mountain ranges as a result of tectonic pressure brought on by the collision of continents between India and Eurasia (Powell, 1979; Valdiya, 2016; Rehman *et al*., 2017; Ali *et al*., 2019). This area was defined as the Himalayan foreland basin. This marginal basin stretches east-west from Nepal to Pakistan for almost two thousand (2000) kilometers (Ali at el., 2019 Fig. 1d). During the Neogene times, the foreland basin had received huge amount of sediments, produced in response to on-going India-Eurasia collision, and materialized as distinctive individuality known as "Siwaliks" (Najman, 2006).

Three sub-groups comprise the rocks of Pakistan's Siwalik Group: Lower, Middle, and Upper (Shah, 2009; Ali *et al*., 2020a). Lower Siwalik includes the Kamlial & Chinji Formations, which are characterized by mudstone-dominated facies over sandstone. The Nagri and Dhok Pathan Formations, which are mostly arenacious in nature with a characteristic alternation of sandstone/mudstone facies, are the names given to the mid-Siwalik formations. The Upper Siwalik Soan Formation is mostly characterized by conglomeration. A series of Himalayan foreland Fold-and-Thrust-Belts was created by the ongoing continental collision between Eurasia and India, as well as the periodic southward propagating over-thrusting and folding of the Indian plate's crustal blocks (Blisniuk *et al*., 1998). Among these, the Surghar-Shingar Range (Fig. 1c) is an arcuate mountain band that is situated west of the Indus River and is displaced by the active dextral Kalabagh Fault (Fig. 1c) situated as the western extension of the Salt Range, west of the Indus River, and symbolizing the outermost Himalayan mountains (Powell, 1979; Fig. 1b). As they border the Bannu Basin (Fig. 1c) to the west, the SSR exhibit an EW pattern along the southern edge of the Kohat Plateau and achieve NS structural style (Khan and Opdyke, 1987b; Rehman *et al*., 2017; Ali at el., 2020a).

According to Akhtar (1983), this SSR is an asymmetrically overfolded anticline that exposes Paleocene and Mesozoic rocks in the core that are underlain by Permian strata. Older rock units outcrop on the eastern side of the current research area, whereas rocks from the Siwalik Group outcrop on the western limb. While Permian and Mesozoic rocks are in contact in the north, the Surghar Thrust, which is equivalent to the Salt Range Thrust and may be present along the axis of the Surghar anticline, has brought Punjab foreland alluvium and Neogene rocks into contact with each other in the south (Gee, 1989).

The range has undergone several orogenic processes, which led to stream erosion in a head-ward direction, the development of rough topography expression, and the formation of precipitous cliffs. The resulting debris was deposited on the nearby Indus-Bannu plains (Ali *et al*., 2020a). Rocks from the Siwalik Group began to deposit, marking the end of the marine deposition period in the SSR. The existence of a significant, distinct conglomeratic deposit that is mostly composed of Eocene pebbles and boulders marks the limit of Lower Siwalik (Azizullah and Khan, 1997).

The Siwalik Group rocks exposed on the western limb of the SSR have a thickness of 5300 m, whereas the Dhok Pathan Formation has a thickness ranging from 807 to 1540 m. The sequences of sandstone and shale alternate cyclically in an upward fining pattern (Ali *et al*., 2019). According to Khan and Opdyke's (1987b) magneto-stratigraphic research, the Dhok Pathan Formation in the studied region is 7.5–2.5 Ma.

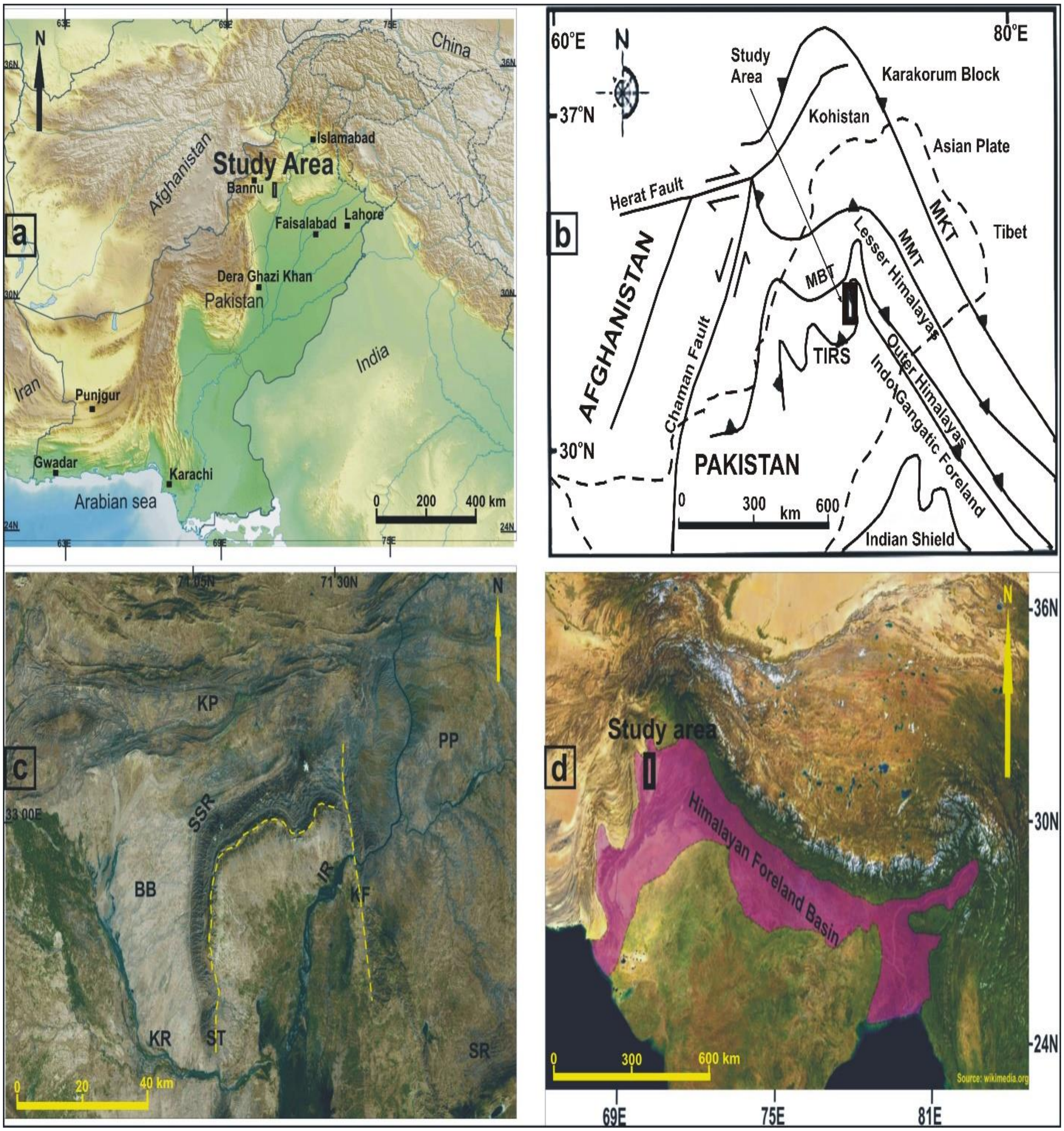

Figure 1: a) Regional map of Pakistan depicting location of study area, b) Tectonic map of NW Pakistan, c) Zoom earth satellite image of SSR and surrounding areas. BB: Bannu Basin, PP: Potwar Plateau, KP: Kohat Plateau, KF: Kalabagh Fault, IR: Indus River, KR: Kurram River, SR: Salt Range, ST/SF: Surghar Thrust/Fault, SSR: Surghar Shingar Range, MKT: Main Karakoram Thrust, MMT: Main Mantle Thrust, MBT: Main Boundary Thrust, MFT: Main Frontal Thrust, TIRT: Trans Indus Range Thrust, d) Himalayan foreland basin (purple color) (after Ali et al., 2019).

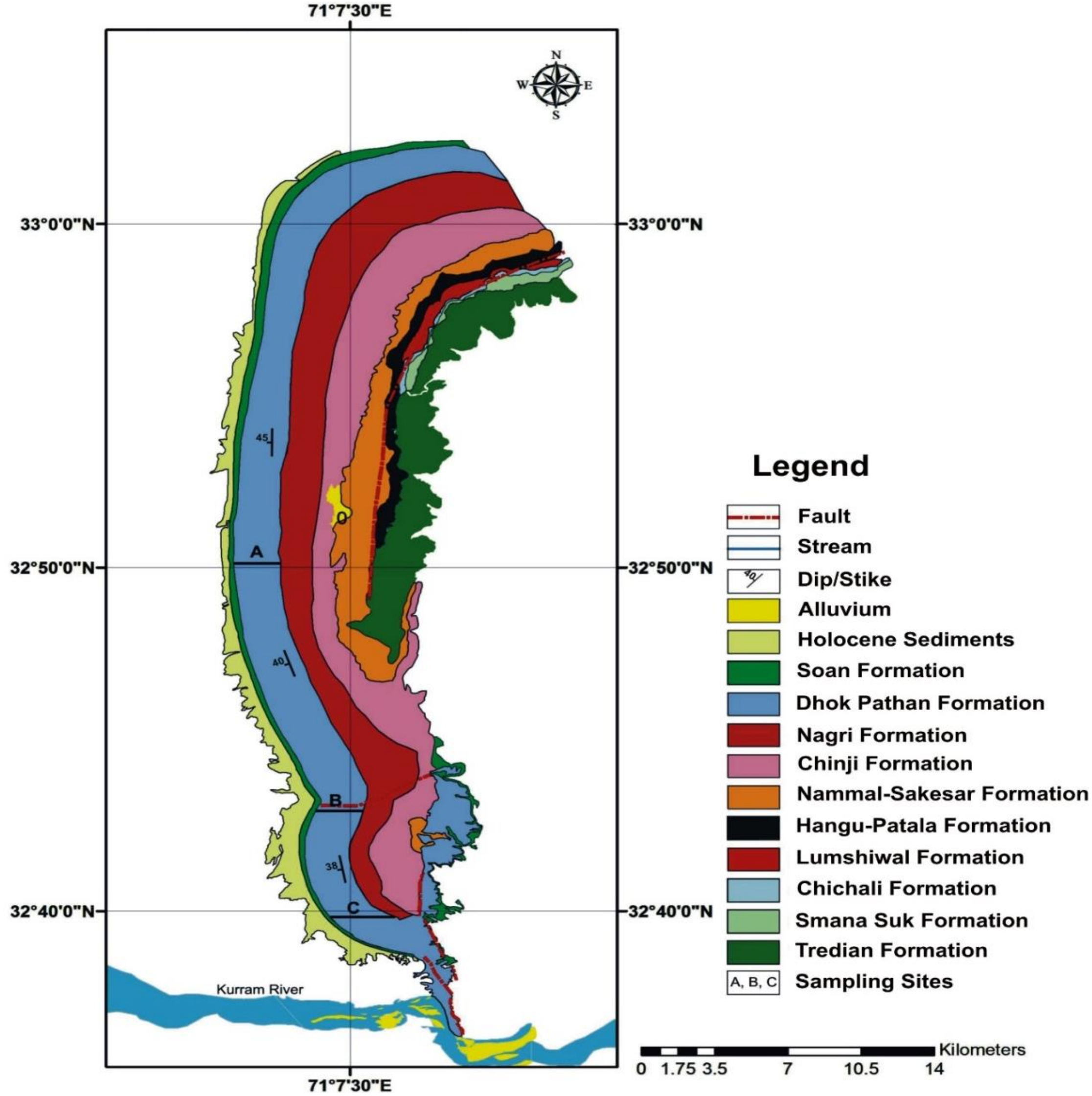

Figure 2: Detailed geological map of NS segment of SSR showing location of sampling sites (after Ali et al., 2019)

MATERIAL AND METHODS

The summary of methods used to analyze the samples is as following: -

i. From the sampling sites marked A, B, and C in figure 2, ten (10) drill core samples and twelve (12) surface samples of sandstone containing organic matter were taken. Innovative methods were employed to extract organic materials from sandstones. Impurities including carbonate were eliminated by boiling a 0.5M HCl solution. After a day, the identical procedure was carried out again. To confirm that all carbonates had been removed, the samples were treated once again with HCl solution after being rinsed or washed with distilled water. Samples were then rinsed or cleaned with distilled water and dried in an oven. To prevent any contamination, the samples were ground to a 200µm size using a manual mortar and pestle made of non-iron.

ii. The elemental analyzer isotope mass spectrometer (EA-IRMS) was utilized to evaluate the samples in order to get the $\delta^{13}C$. Ten (10) representative samples of organic matter were subjected to these examinations.

iii. Following the preparation of polished slides, the categorization, characterization, and morphological features of organic matter were examined using a plain polarized microscope, reflected light microscopy, and scanning electron microscopy (SEM, Nova Scan 450). To prepare samples for SEM analysis, gold coating was applied. The Massoud and Kinghorn (1982) and Hart (1986) categorization systems used as a point of reference.

The samples were examined at the East China University of Technology, Nanchang's "State key Laboratory Breeding Base of Nuclear Resources and Environment" and the "Jiangxi Province Key Laboratory of the Causes and Control of Atmospheric Pollution." China.

RESULTS

4.1 Characterization of Organic Matter: Woody tissues or coaly material, exhibiting a range of colors from grey to light-brown to black, revealed the distinctive cellular structure (Fig. 3a & 3b). The morphology obtained by SEM

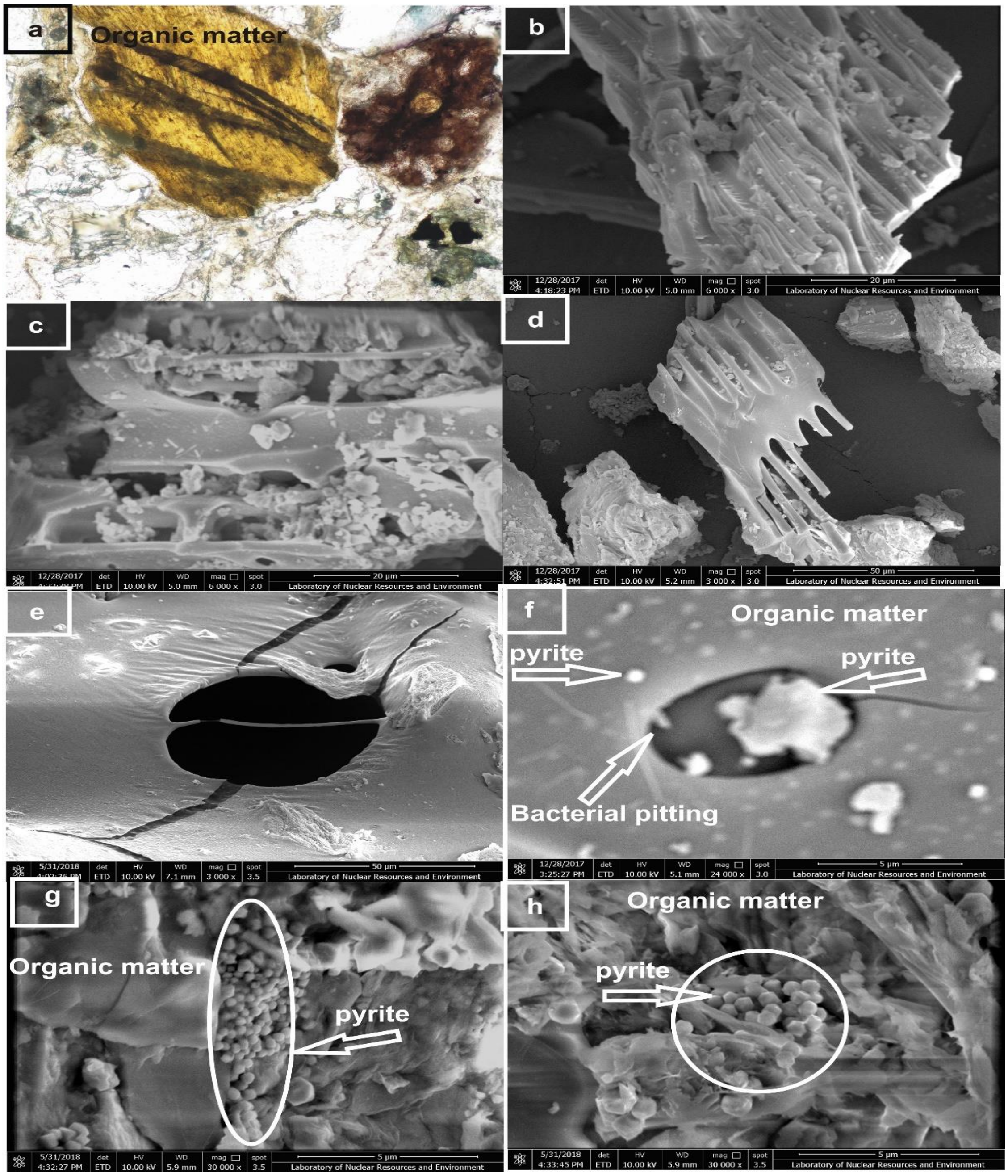

Figure 3: Photomicrograph and SEM back scattered images of organic matter, a) coaly phytoclast plain polarized light, b) back scattered image of phytoclast, c & d) fungal and bacterial attack on phytoclast, e) bacterial pitting and scaring on organic matter, f) replacement of organic matter with pyrite, g & h) development of pyrite crystals in interstitial spaces of organic matter.

imaging indicates that these organic materials have a robust structural framework and an angular form. However, the cell walls have been somewhat disturbed by fungal attack (Fig. 3c & 3d). Furthermore, replacement characteristics including scarring, pyritization, and bacterial pitting were also noted (Fig. 3e, 3f, 3g & 3h). According to Massoud and Kinghorn (1982) and Ercegovac and Kostic (2006), these phytoclasts can be classified as huminite/vitrinite or as amorphous structural to weakly maintained phytoclasts (Hart, 1986). These phytoclasts are categorized as Type-III kerogen and are typically mainly provided by terrestrial plants (Massoud and Kinghorn, 1982).

$\delta^{13}C$ isotope: The average $\delta^{13}C$ value (n = 10) was -26.56‰, with a range of -24.50 to -28.43‰. The tested organic material's total carbon content varied from 15.9 to 49.55%, with an average value of 33.87% (n = 10). These estimated $\delta^{13}C$ values corresponded to terrestrial plants of the C_3 type (Ehleringer, 1989). The ^{15}N (nitrogen) & $\delta^{18}O$ (oxygen) values were could not detected as beyond the detection limits of the instrument. The results are tabulated in Table-1.

Table - 1: EA-IRMS analytical results of organic matter from sandstone samples of Dhok Pathan Formation.

Sr. No.	Sample No.	$\delta^{13}C$ ‰	Total C %	Sample type
1	211	-26.15	35.42	sandstone
2	212	-25.23	45.95	sandstone
3	215	-26.64	15.82	sandstone
4	216	-28.43	31.80	sandstone
5	217	-28.26	37.75	sandstone
6	218	-27.28	36.19	sandstone
7	219	-24.50	33.41	sandstone
8	220	-27.85	36.44	sandstone
9	2122	-24.70	49.55	sandstone
10	2152	-28.80	15.96	sandstone
Average $\delta^{13}C$ = -26.56‰, Average total Carbon = 33.87%				

DISCUSSION

Three distinct classes of carbon isotopes are displayed by plants. C_3 plants include almost all tree species (climate independent), shrub, herb, and grass genus that prefer a cooler growing season (Quade *et al*., 1989). Their average $\delta^{13}C$ value is -27‰, but can range from -35‰ to -20‰ depending on parameters such as moisture stress, plant type, lifespan, and light intensity (Ehleringer, 1989). Certain shrubs from the Euphorbiaceae and Chenopodiaceae families that thrive in warm climates are classified as C_4 plants. The average $\delta^{13}C$ for these species is around -13‰. The term "crassulacean acid metabolism" (CAM) refers to lustrous cacti and yuccas that are not very important to the ecology outside of deserts (Quade *et al*., 1989). Changes in climate and the movement of continents and oceans over the Neogene period had profound and varied impacts on Earth's biota (Barry *et al*., 1985, Ali at al., 2018). The majority of biomass consisted of pure or almost pure C_3 prior to 7.4-7.0 million years ago (Quade *et al*., 1989). The majority of Pakistan's vegetation during the mid-to late-Miocene was composed of C_3 grasses, based on isotopic and microwear data reported by Morgan *et al*. (1994). Between 8 and 6 million years ago, there was a notable rise in C_4 biomass in four widely separated regions: East Africa, South America, low-latitude North America, and Pakistan (Cerling *et al*., 1997). The Himalayan foreland basin was mostly covered by C_3 vegetation-type from the late Miocene to the early Pliocene, based on $\delta^{13}C$ evidence from the Dhok Pathan Formation (Ali at al., 2019).

The amount to which organic matter is changed is influenced by the surface surroundings to which it is exposed. An accelerated rate of organic matter degradation may result from extended exposure. Anaerobic conditions resulted in the breakdown of organic materials into H_2S, CH_4, CO_2, and NH_3, while aerobic conditions transformed them into H_2O, CO_2, SO_4^{-2}, NO^{-3}, etc. (Ehrlich, 1981). If these degradative processes were successful, the organic matter would either remain in the water column as a component of the dissolved chemical cycle or be deposited at the interface between sediment and water (Hart, 1986). The origin of the organic matter is irrelevant to the biological, physical, and chemical processes that must occur upon deposition at the sediment-water interface.

Geologically speaking, the depositional habitats must be understood at the depths below, when the redox-potential discontinuity, or Eh, becomes negative. These environments cannot be found at the sediment-water interface or in the highest layers of a deposit (Hart, 1986). In fresh water environments ranging from low oxygen to low anoxicity, within an active river channel with relatively high energy, the light-brown, grey-to-blackish coaly material/woody tissues (phytoclasts) from the Dhok

Pathan Formation sandstones are generally preserved, according to Ercegovac and Kostic (2006). Phytoclasts with a robust structural framework and an angular morphology have the potential to biodegrade lingo-cellulosic materials in oxic conditions. Evidence of biodegradation includes fungal attack, pitting, and scarring caused by bacteria. According to Hart (1986), this breakdown can happen in environments with or without oxygen. Signs of bacterial pitting suggest that deterioration occurred in anoxic environments. This organic matter has the potential to transform into inertinite when exposed to oxidizing conditions and temperatures ranging from high to normal (Ercegovac and Kostic, 2006). Due to the complexity of the diagenetic processes, the diagenetic products are not always easy to comprehend or relate to their parent materials. Due to the fact that diagenetic and depositional controls are likely exceedingly variable, lacking clear trends, and reactions are seldom in equilibrium, it is more challenging to comprehend the early diagenetic modification processes (Ragland *et al*., 1979; Swart, 1984). Possible subtle but noticeable indicators of degree of alteration include stream hydrology, organic matter solubility, and the sediment-water interface.

Frambiodal and other forms of pyrite can partially replace organic matter under specific diagenetic conditions. The rate of sediment accumulation determines the pyritization process, which in turn determines the link between metabolizable organic matter flux and sulphate reduction (Fisher and Hudson, 1987). As a diagnostic tool for certain environments/post depositional conditions, sulphate-reducing bacteria can facilitate this pyritization process (Brand, 1994). The presence of organic matter in sediments suggests that microorganisms were alive and healthy, that essential metals like iron and sulfur were present, and that redox conditions were dominant during the surface burial process (Howarth, 1979; Raiswell and Berner, 1985). Dyoxic conditions had to be prevailed upon in order to preserve this pyrite-organic matter replacement mechanism. The SEM images of organic matter from the Dhok Pathan Formation sandstones clearly show the presence of frambiodal and other forms of pyrite as a replacement or diagenetic element. This section of the Dhok Pathan Formation was found to have been deposited in an environment with low oxygen levels.

Conclusion: In the studied part of the NW Himalayan foreland basin, C_3 vegetation-type was the most prevalent throughout Neogene periods, according to morphology and $\delta^{13}C$ isotopic investigations of organic matter. The organic matter, which is categorized as Type-III kerogen, was mostly provided by terrestrial land plants. The phytoclasts, which come from terrestrial plants, are classified as huminite/vitrinite. The Dhok Pathan production's sandstones were most likely formed in anoxic environments, as evidenced by the occurrence of fungal assault, bacterial activity-induced pitting and scarring, biochemical degradation, and the replacement/diagenetic production of pyrite.

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